MTL TR 89-41

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# EFFECT OF ANNEALING CONDITIONS ON THE STRUCTURAL AND SUPERCONDUCTING PROPERTIES OF Y-Ba-Cu-O FILMS

JAE RYU, YIZHOU HUANG, and CARMINE VITTORIA
NORTHEASTERN UNIVERSITY

DANIEL F. RYDER, Jr.
TUFTS UNIVERSITY

JAMES V. MARZIK
U.S. ARMY MATERIALS TECHNOLOGY LABORATORY
MATERIALS SCIENCE BRANCH

RICHARD BENFER and WILLIAM A. SPURGEON
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May 1989



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| 4. TITLE (and Subtitle)<br>EFFECT OF AN... ...CONDITIONS ON THE STRUCTURAL ... ...PERCONDUCTING PROPERTIES OF Y-Ba-C... ...LMS | | 5. TYPE OF REPORT & PERIOD COVERED |
| | | 6. PERFORMING ORG. REPORT NUMBER |
| 7. AUTHOR(s)<br>Jae R...,* Yizhou Huang,* Carmine Vittoria,* Daniel F. Ryder, Jr.,† James V. Marzik, Richard Benfer, and William A. Spurgeon | | 8. CONTRACT OR GRANT NUMBER(s) |
| 9. PERFORMING ORGANIZATION NAME AND ADDRESS<br>U.S. Army Materials Technology Laboratory<br>Watertown, Massachusetts 02172-0001<br>ATTN: SLCMT-EMS | | 10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS<br>D/A Project: 1L161102AH42 |
| 11. CONTROLLING OFFICE NAME AND ADDRESS<br>... Army Laboratory Command<br>... Powder Mill Road<br>...phi, Maryland 20783-114... | | 12. REPORT DATE<br>May 1989 |
| | | 13. NUMBER OF PAGES<br>11 |
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| | | |
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| Superconductors | Electrical properties | Ceramic oxide |
| Ceramic materials | Oxides | |
| Thin films | Electroceramics | |

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Block 18. Continued

To be published in Proceedings of the Conference on the Science and Technology of Thin Film Superconductors, Colorado Springs, CO, Nov. 14-18, 1988.

Block 20.

## ABSTRACT

The effect of various annealing treatments on ion beam sputter-deposited $Y_1Ba_2Cu_yO_{7-x}$ (y=3 and 5) amorphous thin films has been studied using scanning electron microscopy (SEM), X-ray diffraction and dc resistivity measurements. SEM analysis showed that the morphology of the films were clearly divided into three categories: an equiaxial grain structure, a well-oriented grid-like microstructure and a textured platelet structure. The equiaxial grain structure (random orientation according to X-ray diffraction measurements) was obtained when the film was annealed at slow heating and cooling rates in an oxygen atmosphere. The grid-like microstructure (b/c-axes oriented normal to the plane) was obtained in Cu-rich composition (y=5) by rapid thermal annealing. A textured platelet microstructure (c-axis oriented normal to the plane) was obtained using slow heating rates and by carefully controlling the gas environment at the temperature region near the orthorhombic-tetragonal transition. Thus, we have demonstrated the ability to control the morphology and orientation of the film by varying the annealing process.

# EFFECT OF ANNEALING CONDITIONS ON THE STRUCTURAL AND SUPERCONDUCTING PROPERTIES OF Y-Ba-Cu-O FILMS

J. Ryu[a], Y. Huang[a], C. Vittoria[a], D. F. Ryder, Jr.[b], J. Marzik[c], R. Benfer[c], and W. Spurgeon[c]

a: Northeastern University, Center for Electromagn. Research, Boston, MA 02115
b: Tufts University, Laboratory for Materials and Interfaces, Medford, MA 02155
c: Army Materials Technology Laboratory, Watertown, MA 02912

## ABSTRACT

The effect of various annealing treatments on ion beam sputter-deposited $Y_1Ba_2Cu_yO_{7-x}$ (y=3 and 5) amorphous thin films has been studied using scanning electron microscopy (SEM), x-ray diffraction and dc resistivity measurements. SEM analysis showed that the morphology of the films were clearly divided into three categories: an equiaxial grain structure, a well oriented grid-like microstructure and a textured platelet structure. The equiaxial grain structure (random orientation according to x-ray diffraction measurements) was obtained when the film was annealed at slow heating and cooling rates in an oxygen atmosphere. The grid-like microstructure (b/c-axes oriented normal to the plane) was obtained in Cu-rich *composition (y=5) by rapid thermal* annealing. A textured platelet microstructure (c-axis oriented normal to the plane) was obtained using slow heating rates and by carefully controlling the gas environment at the temperature region near the orthorhombic-tetragonal transition. Thus, we have demonstrated the ability to control the morphology and orientation of the film by varying the annealing process.

## INTRODUCTION

The discovery of superconductivity above liquid nitrogen temperature in oxide ceramics has ignited intense research on these materials throughout the world. For planar device applications, such as microwave and millimeter wave components, high power electromagnetics, and high speed electronic devices, it is essential to produce thin superconducting films. Furthermore, it is now well known that the superconducting properties of these materials are highly anisotropic.[1] Therefore, it is important to be able to control the orientation and morphology of these films. Although preferentially oriented polycrystalline films[2,3] and single crystalline films[4,5] have been reported by several groups, it is not yet clear what the determining factors are in controlling film orientation. In the present study, we report the annealing processes by which we have been able to produce thin films having several unique morphologies and orientations.

Futhermore, we report here the effect of microstructure on the superconducting, properties, wet chemical etching rate, and the stability of this film to the laboratory environment.

## EXPERIMENTAL PROCEDURE

Films of YBaCuO were deposited by using an ion beam sputtering technique with sintered single oxide targets. The nominal compositions of the targets were Y=1, Ba=2.3 and Cu=3.8 or 6. The compositions of films measured by electron probe microanalysis (EPMA) were $Y_1Ba_2Cu_3O_{7-x}$ and $Y_1Ba_2Cu_5O_{7-x}$ using $YBa_{2.3}Cu_{3.8}O_{7-x}$ and $Y_1Ba_{2.3}Cu_6O_{7-x}$ targets, respectively. Usually, a 1mm thick film was deposited on single crystalline (100) oriented cubic zirconia (YSZ), $SrTiO_3$ or MgO substrates in 1hr. All films were deposited without externally heating the substrate. As-deposited films had an amorphous structure. The films were then annealed using various heating and cooling cycles, as shown in Fig. 1. In process A, the films were heated under flowing oxygen (20sccm) in a tube furnace to 1093K-1143K in 3hrs and held at this temperature for 1-4hrs and subsequently cooled to room temperature in 10-18hrs.

Process B involved three steps. In step one, the films were rapidly heated using a quartz lamp to temperatures ranging from 923K to 973K for 20 min in a 200-500 millitorr oxygen atmosphere and then cooled to room temperature. In step two, the films were heated from ambient temperature to 1173K-1223K in 30sec in a helium atmosphere, held at this temperature for 5-10min. In step 3, the films were cooled in an oxygen atmosphere to 473K over 4 hr. The temperature cycle of process C was identical to that of process A. However, in process C, the heating cycle was performed under helium gas in order to control the orthorhombic-tetragonal transition temperature. The atmosphere was then switched to oxygen for the high temperature hold and subsequent cool down. Surface profillometry, SEM, and thin film x-ray diffraction were used to determine surface roughness, morphology, and orientation of the film, respectively. The dc-resistance of the film as a function of temperature was measured by using a standard four probe technique with pressed indium contacts. The current was 0.1mA in this measurement. For the stability study, the resistance of the film was remeasured after exposure to air for one month. In the chemical etching rate study, the films were etched using a 1:60 mixture of phosphoric acid and DI water.

## RESULTS AND DISCUSSIONS

The SEM micrographs and x-ray diffraction patterns of the films are shown in Fig. 2 and Fig. 3, respectively. A random oriented equiaxial grain structure with grain size around 1mm was observed in films annealed using process A (slow heating and cooling in $O_2$). Films annealed in this process always exhibited a smooth, shiny surface (surface roughness less than 40nm). The texture was independent of the substrate material and slight variations in the Cu concentration. The changes in the film structure during the annealing cycle are complex with several factors to be considered including the amorphous-crystalline transition, the orthorhombic-tetragonal transition, and crystallite nucleation and growth. For process A in which the heating and cooling rates are relatively slow, it is likely that the nucleation rate is much higher than the crystallite growth rate, resulting in a fine grain, randomly oriented structure.

A well oriented grid-like structure with grain size around 1-2mm was observed from the film annealed using process B. The initial rapid heating (process B, step 1) was found to be critical to the formation of this microstructure. In addition, a successful deposition was only achieved using a $SrTiO_3$ substrate.

Films deposited on YSZ and MgO tended to peel away from the substrate during the annealing cycle. Generally, process B was more successful for the films with higher Cu concentration. The surface roughness was determined to be less than 180nm. X-ray diffraction results (Fig. 3b) indicated that the films showed preferred orientation with strong indications of c-axis oriented normal to the plane and weaker orientation normal to the b-axis. Further work is required to determine whether there are two types of grains oriented normal to the (001) and (010) directions, respectively or there exists a single preferred orientation. In the Cu rich film, the existance of a secondary phase, such as CuO and $BaCuO_2$ was observed (as marked (*) in Fig. 3b). Further, in some films annealed by this process, we noticed a presence of an amorphous phase, even after heating at 1193K for 5min. While the average composition of the film was $YBa_2Cu_5O_{7-x}$, as determined by EPMA, the composition of grid area only was $YBa_2Cu_3O_{7-x}$. This result indicated that Cu rich films with the grid-like structure consisted of two phases - stoichiometric composition grid (see G in Fig. 2b), and Cu-rich matrix (see M in Fig. 2c). It has been reported elsewhere[5] that a c-axis oriented epitaxial film of YBaCuO has been produced on $SrTiO_3$ by heating the substrate during deposition at a temperature of 923K. Based on our results and others[4,5], we believe that the well oriented tetragonal crystallites are nucleated at the film-substrate interface during the first step rapid thermal heating at 923K-973K for 20min. These fine crystallites at the interface rapidly grow during the second rapid heating at 1173K-1223K. As a result, stoichiometric composition (as determined by EPMA) grid structure and Cu rich composition islands are formed. In addition, taking into consideration that an excessive amount of Cu enhances the formation of the grid-like structure, the growth rate of the grid may be controlled by the diffusion rate of Cu ion during rapid thermal annealing.

A well oriented platelet structure was obtained in films on the three substrates investigated using process C. At temperatures around 973K-1073K, the annealing gas was switched abruptly from helium to oxygen. The helium gas was introduced during heating in order to control the orthorhombic-tetragonal transition temperature. The surface roughness of the film was less than 300nm, and the platelet-like grains were approximately 20mm in size (Fig. 2c). The film showed strong preferred orientation with the plane of the film normal to the c-axis as shown by x-ray diffraction (Fig. 3C). It was found that the helium atmosphere during the heating step was critical to the formation of the platelet microstructure. Study on the film-substrate interface using cross section transmission electron microscopy is underway in order to understand the mechanism of nucleation of the crystallite and its effect on the film orientation.

Temperature dependence of the resistance for the films prepared by aforementioned annealing processes are shown in Fig. 4. The resistance (y-axis) is normalized to the room temperature resistance of each film. A $T_c$ onset for film B (grid-like microstructure) and film C (platelet) is the same at 94K, but the zero resistance is observed at 74K and 84K, respectively. A $T_c$ onset and $T_c$ zero for film A (equiaxial grains) is observed at 80K and at 60K, respectively. It is noted that the slope in the resistance curve for film A changes at 180 K, and a slight drop in resistance is observed at 105 K. The broad transition and changes in slope (dr/dK) in the normal state may imply that cation (Cu) ordering is not uniform in the randomly oriented film.[6]

In films with oriented microstructures, there was no degradation in $T_c$ onset, but room temperature resistance increased 5-10% after exposure to air for one month. However, the chemical etching rate (in dilute phosphoric acid) of the oriented

film is 2-3 times higher than that of random orientation film. Chemical etching rate, microstructural, and superconducting properties of the films are summarized in Table 1.

## CONCLUSION

High quality thin films of the YBaCuO superconductor have been produced on YSZ, $SrTiO_3$ and MgO substrates via ion beam sputter-deposition and annealing. The morphology and orientation of the film have been successfully controlled by employing unique annealing processes. Preliminary results indicate that the orientation and morphology of the films are determined primarily by the nucleation rate of crystallites at the film-substrate interface during the initial stage of heating, and the growth rate (or temperature) of these crystallites during high temperature heating. In any case, the lattice matching between the film and the substrate is a key factor in this solid state phase transition. Additional work is required to define the mechanism of nucleation and crystallite growth, and its effect on film orientation. The observed $T_c$ zero resistance is 60K, 74K, and 84K for the film with equiaxial grains, b/c-axis oriented grid-like structure and c-axis oriented platelet microstructure, respectively.

Table 1. Chemical etching rate, microstructural and superconducting properties of the films with various orientations.

| films | structure | room temp. resistivity (mW-cm) | $T_c$ on set (K) | $T_c$ zero (K) | etch rate[a] (mm/min) |
|---|---|---|---|---|---|
| A[b] | equiaxial grains, random oriented | 0.63 | 80 | 60 | 0.154 |
| B[c] | grid-like, b/c-axes oriented | 1.07 | 94 | 74 | 0.909 |
| C[b] | platelet, c-axis oriented | 0.21 | 94 | 84 | 0.309 |

a: Film was etched in a 1:60 mixture of phosphoric acid and DI water at room temperature.
b: On YSZ substrate.
c: On $SrTiO_3$ substrate

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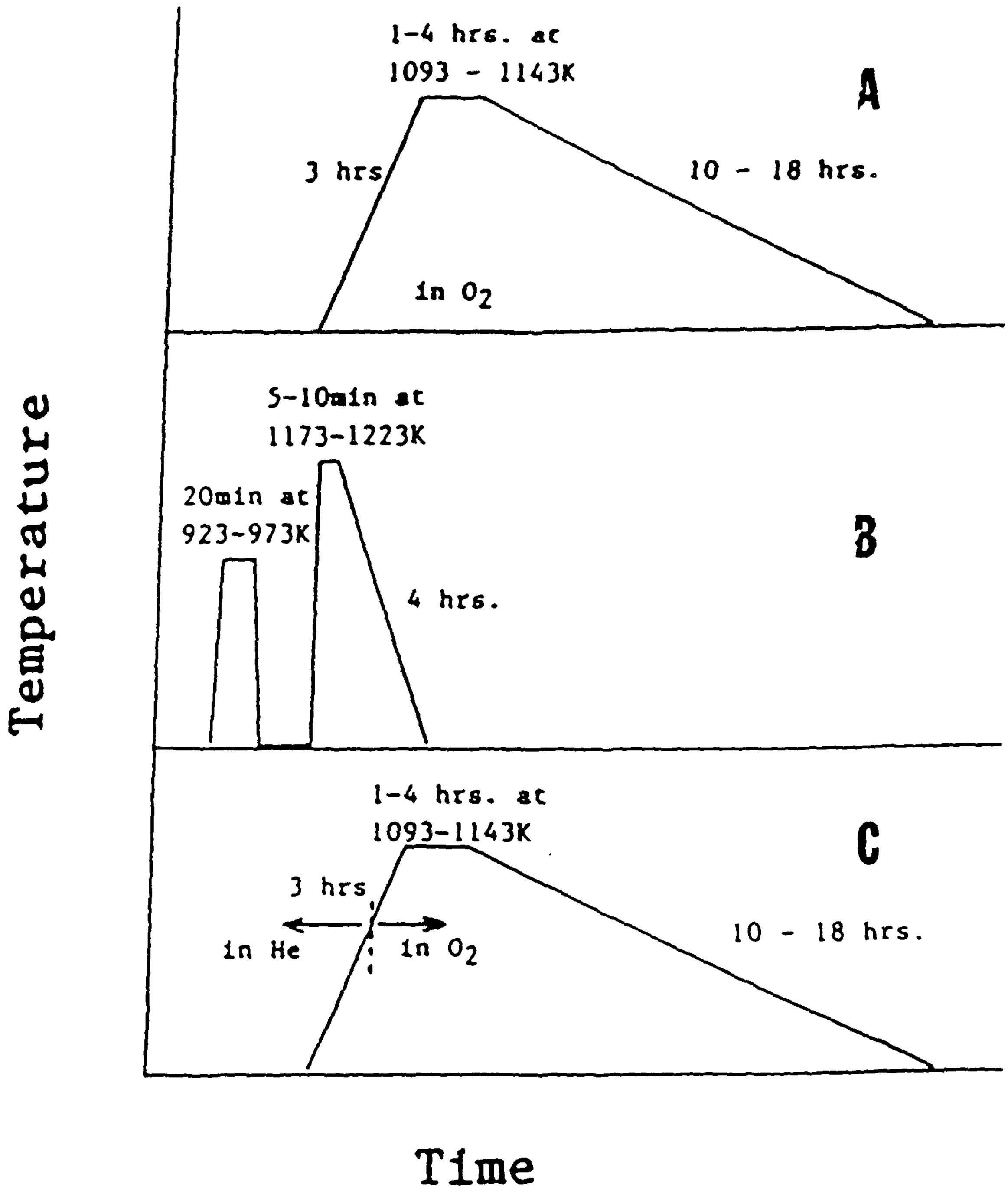
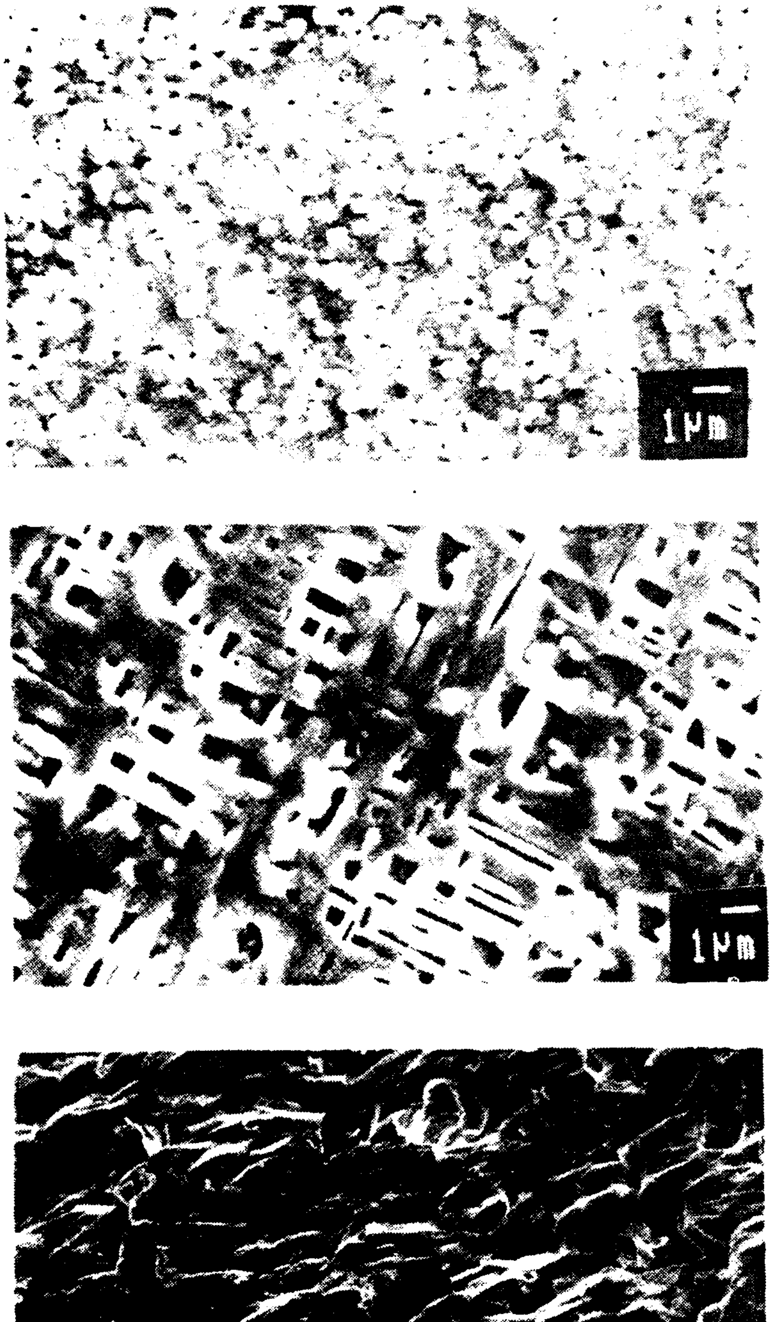



Fig. 1. Annealing procedures used in this research to produce films having a unique morphology and orientation. Note that the temperature-time cycle of process C is identical to that of process A. However, the heating cycle was performed under He gas in process C. Process B involved three steps: in-situ rapid thermal annealing in a low pressure $O_2$; rapid heating in a He atmosphere; and followed by furnace cooling in $O_2$.




(a)

(b)

(c)

Fig. 2. SEM micrographs showing typical microstructures of films with equiaxial grains (a); grid-like structure (b); and platelet (c).

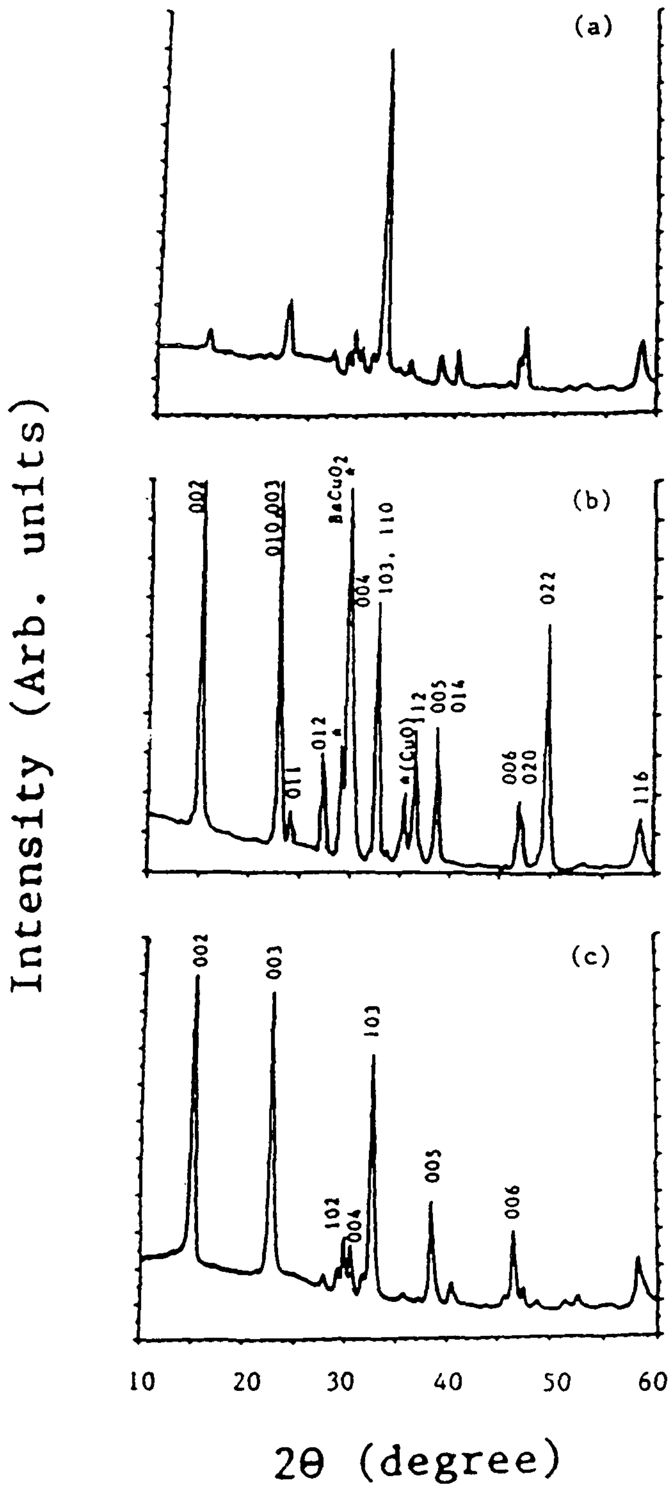



Fig. 3. Thin film x-ray diffraction pattern of the films with various microstructures: (a) equiaxial grain structure - random orientation; (b) grid-like structure - b/c-axes normal to the plane. The peaks from secondary phase are marked (*); (c) platelet microstructure - c-axis oriented normal to the plane.

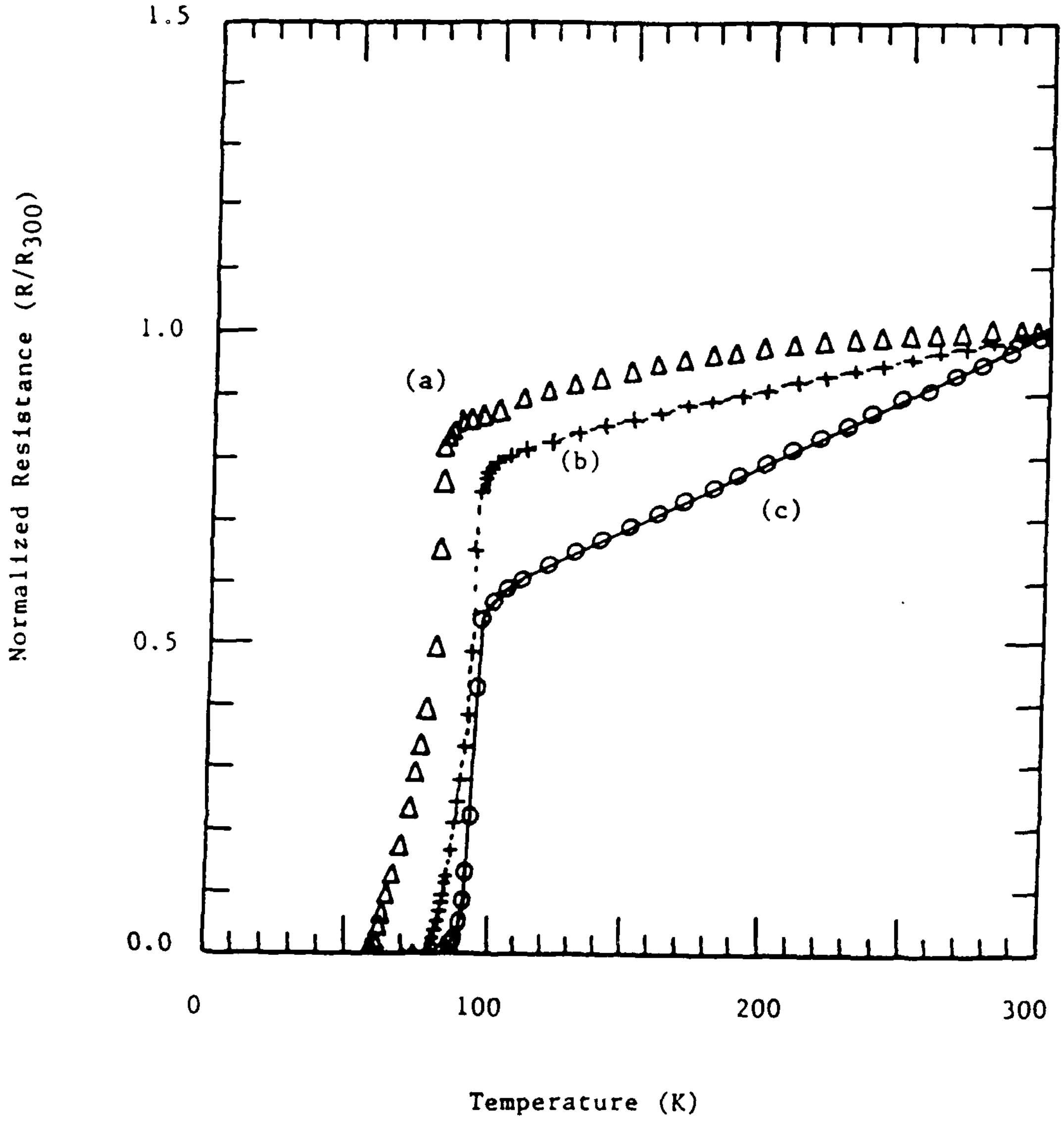



**Fig. 4.** Temperature dependence of the resistance for the film with various morphology and orientations. The resistance is normalized to room temperature resistance ($r/r_{300}$) of each film: (a) randomly oriented film; (b) b/c-axes normal to the plane; (c) c-axis normal to the plane.

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EFFECT OF ANNEALING CONDITIONS ON THE STRUCTURAL AND SUPERCONDUCTING PROPERTIES OF Y-Ba-Cu-O FILMS -
Jae Ryu, Yizhou Huang, Carmine Vittoria, Daniel F. Ryder, Jr., James V. Marzik, Richard Benfer, and William A. Spurgeon

Technical Report MTL TR 89-41, May 1989, 11 pp - illus-tables, D/A Project 1L161102AH42

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The effect of various annealing treatments on ion beam sputter-deposited $Y_1Ba_2Cu_yO_{7-x}$ (y=3 and 5) amorphous thin films has been studied using scanning electron microscopy (SEM), X-ray diffraction and dc resistivity measurements. SEM analysis showed that the morphology of the films were clearly divided into three categories: an equiaxial grain structure, a well-oriented grid-like microstructure and a textured platelet structure. The equiaxial grain structure (random orientation according to X-ray diffraction measurements) was obtained when the film was annealed at slow heating and cooling rates in an oxygen atmosphere. The grid-like microstructure (b/c-axes oriented normal to the plane) was obtained in Cu-rich composition (y=5) by rapid thermal annealing. A textured platelet microstructure (c-axis oriented normal to the plane) was obtained using slow heating rates and by carefully controlling the gas environment at the temperature region near the orthorhombic-tetragonal transition. Thus, we have demonstrated the ability to control the morphology and orientation of the film by varying the annealing process.

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